

**A PROBE TIP AND METHOD OF MANUFACTURING TIPS AND PROBES FOR
DETECTING MICROCURRENT OR MICROFORCE**

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Related Applications

[0001] This application claims priority under 35 U.S.C. § 119 to U.S. provisional application entitled "METHOD FOR MANUFACTURING TIPS AND PROBES FOR DETECTING MICROCURRENT
10 OR MICROFORCE AND DEVICES OBTAINED THEREOF", having Application Number 60/233,276, and filed on September 18, 2000. This application also claims priority under 35 U.S.C. § 119 to European patent application number EP 00870246.6, filed on October 27, 2000.

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Background of the Invention

1. Field of the invention

[0002] The invention is generally related to a scanning tunnelling or atomic force microscope and more particularly
20 to the manufacturing of tip and probe used in said scanning tunnelling or atomic force microscope.

2. Description of the Related Art

[0003] Scanning tunnelling microscopy (STM) and electrical
25 atomic force microscopy (AFM) enables us today to carry out electrical and/or topographical measurements on modern semiconductor devices with nanometer (nm) spatial resolution. These measurements are done by scanning at least one small conductive tip over the device surface and recording at the
30 same time topographical information and/or electrical signals.

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[0004] Metal is preferably used as tip material due to its excellent electrical conductivity. Silicon (Si) tips with a thin metal coating, typically 10-25 nm thick, are commercially available since the early 1990's but the metal coatings rapidly wear off. Therefore solid metal pyramids made by the so-called moulding technique have been developed which are integrated into Si cantilevers or metal cantilevers. The metal cantilever probes, also called full metal probes, are preferred because they are highly conductive and require fewer process steps. In European Patent No 0899538 "A probe tip configuration, a method of fabricating probe tips and use thereof", (which is hereby incorporated by reference in its entirety) the inventors discloses a method of fabricating probe tips applying said so-called moulding technique. The fabrication and use of a metal tip is disclosed in "Fabrication and use of metal tip and tip-on-tip probes for AFM-based device analyses", by T. Hantschel et al. Proc. of SPIE, 3512, pp. 92-103, 1998 (which is hereby incorporated by reference in its entirety). A method of manufacturing full metal probes is disclosed in "Fabrication of an all-metal atomic force microscope probe", J.P. Rasmussen, Proc. Of Transducers '97, p 463-466, 1997 or in "The fabrication of a full metal AFM probe and its applications for Si and InP device analysis", T. Hantschel et Al., Proc. Micromachining and Microfabrication conference, 1999 (both hereby incorporated by reference in their entirety). This manufacturing method is schematically illustrated in figure 1e-1f. In figures 1e and 1f the cantilever is drawn in side view for the purpose of teaching.

[0005] The above mentioned process method suffers from the following disadvantages:

1. The wafer needs to be processed from both sides whereby precautions have to be taken to protect the non-processed side.
2. A lithographic tool, e.g. a mask aligner, is required to align patterns defined on the backside of the substrate to the front or topside of the wafer.
3. The backside of the silicon substrate will be etched to release at the front-side the cantilever with metal tip (see fig. 1a,e) and hence the substrate cannot be re-used.
4. This backside etching requires wafer scale uniformity of e.g. wafer thickness, etch speed, etch selectivity to avoid local over-etching of the tip and/or probe and it is a time consuming step.
5. During this etch step the tip, e.g. its radius or sidewalls, can be modified by the etch chemicals.
- [0006] In European Patent No. 0763844 entitled "method of manufacturing micro-tip and female mould substrate therefor, and method of manufacturing probe with micro-tip" (which is hereby incorporated by reference in its entirety) some of these disadvantages are indicated. The inventor T. Yagi proposes a method to overcome some of the above-mentioned disadvantages, as illustrated in figure 2. According to European Patent No. 0763844 the first substrate is used to define the probe tip (23) while the second substrate is used to define the cantilever. Depending on the choice of the second substrate or its stack composition several types of cantilevers can be obtained. The first substrate could be re-used.
- [0007] The above mentioned process method still suffers from the following disadvantages:
1. Two substrates are required to manufacture a probe tip with cantilever.

2. The metal, peeling layer and substrate have to be chosen such to obtain the correct relative strength of the different layers to allow tearing off the first second substrate from the probe tip.
- 5 3. The second substrate still requires an etching of the rear surface to define a cantilever and to release the probe tip. All problems related to such rear side etching remains.

Therefore, there is a need in the industry for a cost
10 effective method of manufacturing tips and probes for use in scanning tunnelling or atomic force microscopes.

Summary of the invention

[0008] It is an aim of the invention to achieve a cost and
15 time efficient method to manufacture probes used in scanning microscopy, e.g. AFM, STM, SSRM or likewise. This method comprises front side processing steps and avoids the backside lithography and time consuming etching steps required to create a probe membrane or to release the probe membrane.
20 This method requires one substrate to form a full probe consisting of a probe tip, cantilever and probe membrane. This method allows easy removal of the probe prior to mounting the probe into a scanning microscope.

[0009] Another aim of the invention is to achieve a
25 method of manufacturing a full metal probe comprising front side lithography steps, thereby allowing the probe tip and the cantilever to be separately processed. Hence the probe tip can be made from a material or metallisation scheme different from that of the cantilever. The characteristics
30 of the probe tip can optimised independently from the cantilever.

[0010] It is one object of the invention to disclose a cost and time efficient method to manufacture probes used in scanning microscopy, e.g. AFM, STM, SSRM or likewise. This method comprises front side processing steps and avoids the
5 backside lithography and time consuming etching steps required to create a probe membrane or to release the probe membrane.

[0011] One object of the invention is to a method comprising the steps of providing a substrate, depositing on
10 said substrate a hard mask. During a first patterning step a mould is created in said hard mask and said substrate. A first layer is deposited on said patterned hard mask. This first layer has a sufficiently high enough adhesion to said hard mask to allow further processing, but this adhesion
15 force is weak enough to be overcome when peeling the probe gradually from said underlying hard mask. During a second patterning step said first layer is patterned to form a probe tip configuration comprising at least a probe tip. By partially or fully under-etching said probe tip configuration
20 from the front side of the substrate, the probe tip configuration can be removed from said substrate by lifting the probe.

[0012] Said method can further comprise the step of forming selective to said patterned first layer at least one
25 additional layer before under-etching said patterned stack.

[0013] Said method can further comprise the step of mounting a holder chip to said probe tip configuration before peeling of the probe.

[0014] Said hard mask can be nitride, oxide, oxynitride
30 or a combination of layers. Said first layer can be a metal, preferably a stack of Ti:W and Au.

[0015] Another object of the invention is to a method of manufacturing a full metal probe comprising front side lithography and processing steps, thereby allowing the probe tip and the cantilever to be separately processed. Hence
5 the probe tip can be made from a material or metallisation scheme different from the cantilever. After depositing the first layer or a stack of layers the area of the probe tip is defined in an intermediate, third, patterning step. Hereafter a second layer can be deposited on said hard mask
10 and said patterned first layer. This second layer will be patterned in the last patterning step to define the probe tip configuration consisting of probe membrane or contact area, cantilever and overlapping the probe tip area. Said second layer can be a metal, preferably a stack of Ti:W
15 and Au.

[0016] Said method can further comprise the step of forming selective to said patterned second layer at least one additional layer before under-etching said patterned stack.

20 Brief Description of the Drawings

[0017] All drawings are intended to illustrate some aspects and embodiments of the invention. Devices and fabrication steps are depicted in a simplified way for reason of clarity. Not all alternatives and options are shown and
25 therefore the invention is not limited to the content of the given drawings. Like numerals are employed to reference like parts in the different figures.

[0018] Figure 1 represents a prior art method of manufacturing a full metal probe comprising a tip, cantilever
30 and membrane applying the moulding technique, illustrated by cross sectional views

- [0019] Figure 2 represents a prior art method of manufacturing a tip to avoid backside etching of the moulding substrate, illustrated by cross sectional views
- [0020] Figure 3 represents a process scheme for peel-off probes made by two lithography steps, upper part is top view, and bottom part is a cross sectional view along section AA.
- [0021] Figure 4 represents a probe with 4 cantilevers illustrating the peel-off process illustrating the options of fully or partially under-etching the probe
- [0022] Figure 5 represents a probe removal by peel-off according to an embodiment of the invention
- [0023] Figure 6 represents a SEM picture of full metal peel off probe according to the preferred embodiment
- [0024] Figure 7 represents a topography measurements done on a SrTiO_3 calibration sample in tapping mode. Left side shows the AFM picture. Right side shows the measured height h measured along the cross section x .
- [0025] Figure 8 represents a process scheme for peel-off probes with different metallisation scheme for probe tip and cantilever, upper part is top view, and bottom part is a cross sectional view along section AA
- [0026] Figure 9 represents a metal cantilever with hardened tip made by peel off according to one embodiment of the invention.
- [0027] Figure 10 represents I-V measurements in point contact mode using an unhardened Ni tip (a) or a hardened Ni tip (b) according to an embodiment of the invention
- [0028] Figure 11 represents a comparison of unhardened Ni pyramidal tip (a) and hardened Ni pyramidal tip (b), diamond pyramidal tip (c) according to an embodiment of the

invention using SSRM on InP(S) substrate comprising an npn structure.

Detailed Description of the Invention

5 [0029] The following describes the invention in detail in relation to the appended drawings. It is apparent however that a person skilled in the art can imagine several other equivalent embodiments or other ways of executing the invention, the spirit and scope of the invention being
10 limited only by the terms of the appended claims.

[0030] In one embodiment of the invention, it is desirable to apply the method of the invention only to front side processing steps and, more particularly, to lithographic steps. The method allows an easy removal of the probe tip by
15 peeling off the manufactured probe from the underlying substrate.

[0031] In a first embodiment, a method of manufacturing a scanning probe requiring only front side processing steps is disclosed. This method comprises two
20 front side lithographic steps. The peel-off effect is achieved by selectively metallising the area of the probe tip configuration. This metal layer has a sufficiently high enough adhesion to the substrate to allow further processing, but this adhesion force is weak enough to be overcome when
25 peeling the probe gradually from the wafer.

[0032] This method comprises the following steps. During the first patterning step a mould (34a) is formed in the starting substrate (31), preferably a silicon wafer. (fig. 3a) This mould may have one of various shapes, e.g.,
30 pyramidal, a truncated cone, a cylinder. In case a silicon wafer is used a truncated pyramidal etch pit is formed by anisotropically etching the substrate through an opening

defined in a masking layer formed on top of the substrate. Such a masking layer can be a photo resist or a hard mask (32), e.g. nitride, oxide, oxynitride or a combination of layers. This step of defining the probe tip configuration
5 comprises the first front side lithographic step.

[0033] On top of the first surface, i.e. the top surface of the substrate, or the hard mask if used, in which the mould is defined, a first layer (33), preferably metal, is deposited. (fig. 3b)

10 [0034] During the second patterning step the probe tip configuration is defined (fig. 3c). The probe tip configuration consists at least of the probe tip (34), but can also comprise the cantilever (41) and/or the contact area (37) in case a complete probe has to be manufactured.
15 Selective to this first layer additional layers (35) can be deposited using techniques known to a person skilled in the art. If the first layer is a metal additional layers can be plated onto this first layer to create a stack having the desired properties. Such properties are e.g. the mechanical
20 strength of the cantilever that may require a certain thickness, wear-out properties of the probe tip that may require a certain stack composition and hence puts also requirements on the first layer. Between the first layer and the first surface a high contact force exists to allow
25 further processing, but this contact force must be low enough to allow gradually peeling off the probe. Further processing can comprise wet processing steps such as plating, wet etching, cleaning or temperature processing steps such as annealing steps.

30 [0035] After the desired stack is formed the probe tip configuration is fully or partially under-etched (36) (fig. 3d) from the front side of the substrate. If a hard mask is

used, first the hard mask is removed using the probe tip configuration as a masking layer. Underneath the area of the probe tip configuration the hard mask remains. Then the substrate is etched from the front side selective to the probe tip configuration. Partially under-etching will release only the probe tip from the substrate. Fully under-etching will also release the cantilever. Preferably this under-etching is done using an isotropic etch chemistry, e.g. a wet etch such as a KOH-mixture in case of silicon substrates. The probe can now be peeled off from the substrate (fig. 3e).

[0036] A holder chip (53) can be attached to the probe, e.g. to the contact area if formed, to ease further handling or mounting of the probe. If so, a substantial part of the probe tip configuration, comprising the first layer and additional layers formed upon, is temporarily spaced apart from the underlying substrate or hard mask. (fig. 5c) The holder chip is attached to the probe (fig. 5e) and holder chip with the probe is then fully removed from the first surface (fig. 5f).

[0037] In a second embodiment, the method of manufacturing a full metal probe with two front side lithographic steps is disclosed.

[0038] The starting substrates (31) were (100)-Si wafers having a diameter of 150 mm. These wafers were covered with 470 nanometer (nm) thermally grown oxide (32), SiO_2 , and 150nm low-pressure chemical vapour deposited (LPCVD) nitride, Si_3N_4 . (see figure 3a) Photoresist was spun onto the wafers and squares (34) of 40x40 square micrometer (μm^2) were opened in the photoresist during the first lithography step. These squares define the tip area (34). This resist pattern was then transferred to the Si_3N_4 layer

by reactive ion etching (RIE) using a $\text{SF}_6:\text{O}_2$ etching mixture. Next, the resist was removed and the nitride pattern was transferred to the SiO_2 layer by etching the oxide layer with buffered HF (BHF). Inverted pyramids were then etched in 30%
5 KOH at 70°C for 55 minutes. The wafer surface was covered with a 10nm thick layer of sputtered Ti:W followed by a 30 nm thick layer of evaporated Au (33). (see figure 3b)

[0039] Photoresist was again spun onto the wafer. The resist outside the area of the probe tip configuration was
10 removed by the second lithography step, thereby leaving the area of the probe tip configuration covered with resist. The Au layer was etched in a solution of $\text{KI}:\text{I}_2:\text{H}_2\text{O}$. Then the Ti:W layer was removed by RIE in a SF_6 atmosphere. Next, the photoresist was stripped.

15 [0040] A 12 μm thick Ni layer (35) was deposited on the area of the probe tip configuration by electroless Nickel (Ni) plating. Prior to this plating the wafer was pre-treated in a PdCl_2 solution which helps to initiate the autocatalytic Ni deposition. The plating was done in two
20 steps. First, 1 μm of Ni was deposited using the MeTeC-NiP-LT bath from OTB Berlin (Berlin, Germany). The bath temperature was 70°C and the deposition time was 10 minutes. This solution showed in all experiments an excellent starting behaviour but the plated Ni films had a rough surface that
25 would prevent the laser light from being reflected to the detector in the AFM. This first layer is therefore used as a starting layer for the next plating step. The second plating step was done using the ENPLATE Ni 434E bath from Enthone-OMI ('s-Hertogenbosch, Netherlands) at 85°C for 1 hour until its
30 final value of 12 μm . Rough Ni surfaces were smoothed after being plated for some time in the Enthone-OMI solution. After plating the wafer was rinsed in a water bath which had

the same temperature as the plating bath in order to prevent the formation of mechanical stress in the plated Ni film. Rinsing the wafer in cold water immediately after plating resulted in cracks in the Ni cantilevers and the lift-off of
5 plated structures. It should be mentioned that a fresh Enthone-OMI solution had to be used for each plating run. The structures were partially plated if the same bath was used twice. The OTB-Berlin bath showed a good long-term stability and could be used for many runs. (see figure 3c)
10 The selective deposition of the thick Ni films was improved by applying this two-step plating process.

[0041] The Si_3N_4 layer, outside the area of the probe tip configuration, was etched away by RIE in an $\text{SF}_6:\text{O}_2$ atmosphere using the plated Ni film as an etching mask. The
15 SiO_2 layer underneath the nitride layer was opened in BHF. The tip and the cantilever were finally under-etched along the crystal planes (36) in 30% KOH at 70°C for about 5 hours. After a final rinse in hot water at 70°C and careful N_2 drying the probes were ready to be peeled off from the wafer.
20 (see figure 3d)

[0042] Figure 4 illustrates the under-etching of the cantilevers during the anisotropic KOH etching. As can be seen, in this particular design one probe has four cantilevers (41), respectively 200, 400, 600 and 800 μm long.
25 The cantilevers are 65 μm wide and 12 μm thick. The pictures were taken under an optical microscope. Figure 4a shows the probe before etching with the Si_3N_4 layer (32) still in place (Fig. 4a). Figures 4b illustrates the case of under-etching the cantilevers (41), the tips (34) and the bridges (42)
30 (Fig. 4b). Figure 4c shows the probe after the probe membrane was peeled off (Fig. 4c). The yield after under-etching was nearly 100%, which was also confirmed by several

process runs. An advantage of the developed procedure is that the probe membranes are not free standing as the full wafer, contrary to conventional probe moulding, supports them. Therefore the probes survive mechanical shocks during and after processing without any problem. The total under-etching time depends on the longest or largest cantilever.

[0043] Although the probe membranes can be peeled off directly from the substrate, it is more convenient to mount small holder chips to the contact area (37) prior to peeling.

10 The holder chips ease probe handling and mounting in the AFM. Figure 5 shows the peeling procedure schematically. A cross section of the probe tip configuration before peeling can be seen in Fig. 5a. First, one of the two bridges (42, figure 4b) was lifted up with a sharp needle (51) under a microscope

15 (Fig. 5b). After the bridge was broken, the probe membrane (35,33) was gradually peeled off (Fig. 5c). After removing the needle, the membrane fell back into its initial position (Fig. 5d). The connection between the membrane (35,33) and the nitride (32) is no longer present. In one embodiment,

20 the probe remains attached to the substrate via only the unbroken bridge. Silicon chips (53) (3.0 mm long, 1.2 mm wide, 0.6mm thick) metallised with 20 nm Ti:W+45 nm Ni+100 nm Au (52) were then soldered (54) to the probe membranes (35) by a procedure which is described in detail in "Mounting of

25 moulded AFM probes by soldering" , Proc of SPIE 4175, 2000. After soldering, the probe was peeled off completely using a pair of tweezers (51) (Fig. 5f). The whole wafer containing about 600 probes was mounted in this way. By partially peeling off the probe membranes before the soldering step the

30 removal of the probe with holder chip turned out to be very easy and didn't require additional handling.

[0044] Fig. 6a shows a peel-off probe after it was removed from the wafer. It can be seen that the metal membrane (33) is fixed to the Si chip (53) by the solder connection (54). The bright area on the probe membrane is metal which indicates the peel-off area. The dark area is Si_3N_4 from the under-etched area. Fig. 6b shows the end of a full metal cantilever beam. It can be seen that cantilever (41) and metal pyramid (34) are well defined. A close-up of the tip is shown in Fig. 6c. The tips had a radius of typically 50 to 100nm which is slightly higher compared to previous fabricated probes, typically 20 to 50nm. This was due to the long under-etching time of 5 hours where the tip was attacked by the KOH. Figure 7 compares topography measurements on a calibration surface, such as SrTiO_3 , with a Si cantilever with integrated metal pyramid and a peel off probe according to the invention. The metal pyramid on the Si cantilever is clearly sharper than the one on the peel off probe.

[0045] Before peeling off the probe the metal cantilevers are fully under-etched leading to the problems mentioned above. Therefore, the under-etching time in KOH was reduced in the next runs. This was achieved by using shorter cantilevers or peeling off the contact area and the cantilevers instead of under-etching them. The etching time was reduced from 5 hours to 1 hour which corresponds with an etch depth of 40 μm for the given etch solution. The reduced etch time resulted in a substantially reduction of the process time but also in smaller tip radii. AFM or STM microscope with small probe tip radii can measure samples with a higher resolution. This partial under-etching is also shown in figure 4. Figure 4d shows the probe having the probe tip (34) etched free. A schematic cross-section of

figure 4d is drawn underneath the picture. During the last minutes of the etching, the tip is exposed to the etching solution. After cutting one bridge (42), the probe was gradually peeled off as described above. Figure 4e shows the
5 wafer surface after removal of the probe. So if the pyramidal tip (34) is under-etched, the probe with hardened probe tip (34), cantilever (41) and contact (37) area can still be peeled off.

[0046] In a second aspect a method of manufacturing a
10 full metal probe is disclosed comprising front side lithography steps, allowing the probe tip and the cantilever to be separately processed. Hence the probe tip can be made from a material or metallisation scheme different from the cantilever.

15 [0047] In a first embodiment of this aspect the probe tip and cantilever are made of the same material but in a separate and different metallisation scheme.

[0048] It is known in the state of the art that the hardness of as-deposited Ni films made by electroless plating
20 can be increased from about 500HV (HV: Hardness Vickers) up to 1000HV by an annealing the Ni-plated probe tip at 300 to 400°C. Such methods are for example disclosed in "Nickel and chromium plating", 2nd edition, J.K. Denis et al., Butterworths ltd, p. 269-277, 1986, which is hereby
25 incorporated by reference. The plating step and the subsequent annealing step are to be executed after the processing of the probe tip. After applying this procedure on full metal probes a strong bending of the cantilever was however observed, probably due the thermomechanical stress in
30 the cantilever. Therefore the step of annealing the Ni probe tips has to be executed before manufacturing the Ni cantilevers. This separate processing of probe tips and

cantilever was achieved by carrying out two metallisation steps. The overall fabrication process is similar to that described in another embodiment of this invention and therefore only the main steps are discussed.

- 5 [0049] After etching the inverted pyramid (see figure 8a) the probe tip area (34) was metallised by a lift-off procedure. Photoresist was again spun onto the wafer. Applying an additional lithography step will remove the resist substantially above the inverted pyramids. During
- 10 this additional lithography step the probe tip mask of the first lithography step, used to define the probe tip area (34), is used again. The wafer was then covered with 10nm Ti:W+30nm Au (43). In one embodiment, the metal film is structured by lift-off leaving only the probe tip area
- 15 covered with metal, applying techniques known to a person skilled in the art (see figure 8b). The thickness of the metal stack within the area of the probe tip configuration was then increased up to 4µm by exposing the wafer to the Ni plating solution from OTB-Berlin at 70°C for 30 minutes as
- 20 disclosed in another embodiment of the invention (see figure 8c). The pyramidal tip is thus at least partially filled. Afterwards, some tips were hardened by rapid thermal annealing (RTA) for 10 minutes at 300°C. Other tips were annealed at 400°C. Longer anneal times, e.g. 1 hour, can
- 25 improve the hardness of the probe tip. The full wafer surface was then again coated with 10 nm Ti:W+30nm Au (33). In the last lithography step the area of the probe tip configuration (35) was covered with resist and the stack of Au and Ti:W is etched away outside this area (see figure 8d).
- 30 The two-step Ni plating sequence was again applied to metallize the area of the probe tip configuration, as disclosed in another embodiment. The cantilevers (41) were

under-etched (see figure 8e) and the probes were peeled off from the wafer.

[0050] Figure 9a shows the probe topside imaged by an optical microscope after the under-etching was completed. 5 The Ni surface (35) is very smooth and the separately metallised tip area (34) can be clearly distinguished in Figure 9b. The tip area is slightly higher than the cantilever because it was plated twice. Figure 9c shows a metal membrane from the tip side imaged by scanning electron 10 microscopy (SEM). The pyramidal tip shown in the close-up in Figure 9d was annealed at 400°C. The clean side walls indicate that there was no reaction between the Ti:W and the Si during the RTA step.

[0051] In a second embodiment of this aspect the probe 15 tip and cantilever are made of the different materials, each having an appropriated metallisation scheme.

[0052] Although the same metal layers are applied for tip and cantilever in this particular process, the described procedure can also be used to make the tip from materials 20 different from the cantilever material, applying e.g. Cr or diamond to obtain a harder probe tip. The anneal time can depend on the material used and can be further adapted depending on the desired hardness of the probe tip. This does not affect the peeling procedure. The separation 25 between the two metallisation schemes is done by applying an additional patterning step to pattern the probe tip material. Subsequent process steps to improve the quality of the probe tip, such as annealing, can be done after this patterning. Additional material deposition can be done afterwards if the 30 deposition process is selective with respect to the patterned probe tip. Otherwise additional patterning steps are required. As no backside etching is performed the probe tip,

and later on, also the cantilever are supported by the full silicon wafer.

[0053] Figure 10 -11 illustrate the good electrical and mechanical behaviour of probe tips made according to one
5 or more embodiments of the invention.

[0054] I-V measurements on n-type Si ($\rho=0.1 \Omega\text{cm}$) were done in order to find out if the hardened metal tips are able to establish a stable Ohmic contact on Si. It is well-known that an Ohmic contact on Si can be obtained by transforming
10 the Si underneath the tip to the so-called metallic β -tin phase. A pressure of 10-12 GPa is needed for that and the unhardened Ni tips withstood this high pressure for a very short time as can be seen in figure 10a. The first I-V curve (solid line) was collected after increasing the force until
15 the point where the contact behaviour changed from Schottky to Ohmic. The second I-V curve (dashed line) was collected shortly afterwards and showed already an increase in resistance indicating rapid tip degradation at high forces. The electrical contact was lost in the next measurement.
20 Figure 10b shows a set of 20 I-V curves measured using a peel-off probe where the Ni tip was hardened by an annealing step at 400°C for 10 minutes. It can be clearly seen that the hardened probe tip withstands the high forces in point contact and holds the Ohmic regime. The Ohmic regime was
25 also achieved after scanning the tip over the surface for a while and going back into point contact.

[0055] Good results on spreading resistance microscopy (SSRM) on InP device structures have been obtained with metal pyramids integrated into Si cantilevers. A study was done in
30 order to compare the hardness of unhardened and hardened probes with each other (figure 11). Three kinds of Ni tips were tested: unhardened Ni tip (a), Ni tip hardened at 300°C

(b), and metal cantilever with integrated diamond tip (c). The test sample was a multilayer InP structures which was epitaxially grown by metal-organic chemical vapor deposition (MOCVD). The layer structure was p-n-p on a semi-insulating
5 InP(S) substrate where the n-type layer was Si doped and the p-type layers were Zn doped. SSRM measurements were done on a cleaved cross section using a scan size of $10 \times 10 \mu\text{m}^2$ and a scan speed of 1 Hz. The sample was biased at 3.0 V and the same high force was applied to the three probe tips during
10 scanning. No significant difference was found between hardened and unhardened Ni tips. The peel off probes performed as good as the Si cantilever probes manufactured according to the prior art technology.